

PHOTOSYNTHESIS OF CHROMIUM OXIDE NANOPARTICLES FROM CHROMIUM COMPLEXES

HUSSAIN ISMAIL ABDULLAH & LAMYAA JABBAR ABBAS

Department of Chemistry, College of Science, Al-Mustansiriyah University, Baghdad, Iraq

ABSTRACT

Chromium oxide nanoparticles (Cr_2O_3) were synthesized successfully via photosynthesis method. In this technique, chromium oxide nanoparticles were synthesized by using three complexes as a source of chromium, $\text{K}_3[\text{Cr}(\text{C}_2\text{O}_4)_3 \cdot 3\text{H}_2\text{O}]$, $[\text{Cr}(\text{acac})_3]$, and $[\text{Cr}(\text{OC}(\text{NH}_2)_6)\text{Cl}_3]$ without using a surfactant or stirring. The products of Cr_2O_3 nanoparticles were characterized by X-ray diffraction (XRD), Scanning electron microscopy (SEM), Fourier transform infrared (FTIR) spectroscopy and UV-Vis absorption. The results confirm that Cr_2O_3 particles were spherical shape and have a size less than 100 nm. This technique has several features for other techniques of purity, low cost and low particle size.

KEYWORDS: Scanning Electron Microscopy (SEM), Chromium Oxide, UV, Irradiation and Nanoparticles

Received: Dec 03, 2016; **Accepted:** Jan 06, 2017; **Published:** Jan 10, 2017; **Paper Id.:** IJAPBCRFEB20171

INTRODUCTION

Nanoparticles refer to materials whose size in at least one dimension is between 2-99 nm.^[1] Chromium oxide (Cr_2O_3) is the most stable and present in a wide range of temperature and pressure. It is antiferromagnetic when it is small particles, high melting temperature (about 2300°C), has large band gap (~3.4 eV). Furthermore, chromium oxide (Cr_2O_3) is one of the hardest oxide materials.^[2] Therefore, it is important in many applications including: hydrogen storage^[3] catalyst^[4] pigment^[5] coating^[6] wear resistance^[7] sensor^[8] carbon nanotubes^[9] biological applications^[10] solar energy applications.^[11] and advanced digital recording system.^[12] Unlike techniques are available to synthesize Cr_2O_3 nanoparticles such as: chemical precipitation^[13] precipitation gelation^[14] sol gel method^[15] mechanochemical reaction^[16] sonochemical method^[17] novel solvent free method^[18] hydrothermal technique^[19] microwave plasma^[20] thermal decomposition method^[21] microwave refluxing technique^[22]

In the present study a new has been adopted to synthesis chromium (III) oxide NPs by using UV radiation. The UV-irradiation system was used as a source to irradiate the Cr(III) complexes solutions for the purpose of synthesis Cr_2O_3 nanoparticles. Irradiation source which has been used for this purpose was a medium pressure lamp with power of 125 watt which has a maximum light intensity at wavelength at 365 nm. These source have a quartz tube as a checked when we used it as an immersion UV source in the Cr(III) complexes solutions. A test tube was used as a reactor, which contain the Cr(III) complexes solutions. To avoid the rising in temperature emitted from the UV light, the reactor was cooled by ice bath. Figure 1 show this irradiation system.

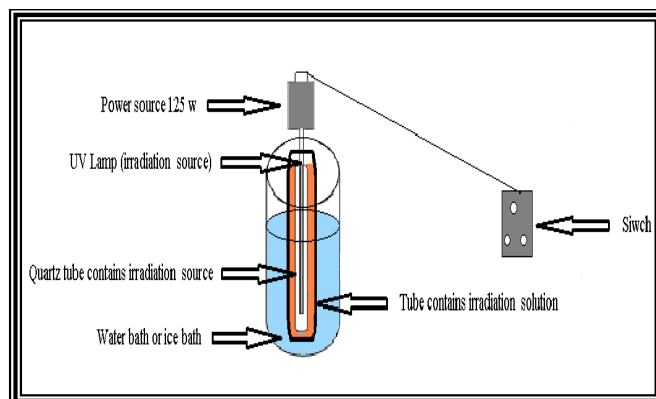


Figure 1: UV- Irradiation System 125 W

Experimental Part

Three complexes were used for the synthesis Cr_2O_3 nanoparticles: potassium chromium oxalate trihydrate solution $[\text{Cr}(\text{C}_2\text{O}_4)_3 \cdot 3\text{H}_2\text{O}]$ and hexakis (urea) chromium(III) trichloride $[\text{Cr}(\text{OC}(\text{NH}_2))_6]\text{Cl}_3$ were prepared according to the literature.^[24] And Cr (III) acetylacetonate $[\text{Cr}(\text{acac})_3]$ was prepared according to the literature.^[25]

Preparation of Solutions

- **From $\text{K}_3[\text{Cr}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}$**

Weight (0.5) g of $\text{K}_3[\text{Cr}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}$ and dissolved in (40) ml deionized water and stirred until a clear blue solution gained.

- **From $[\text{Cr}(\text{acac})_3]$**

Weight (0.5) g $[\text{Cr}(\text{acac})_3]$ and dissolved in (20) ml methanol and stirred until a clear maroon solution gained, then added (20) ml of deionized water to the solution.

- **From $[\text{Cr}(\text{OC}(\text{NH}_2))_6]\text{Cl}_3$**

Weight (0.5) g of the complex and dissolved in (40) ml deionized water and stirred until a clear blue solution gained.

Irradiation Process

- **For $\text{K}_3[\text{Cr}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}$**

A (40) ml of $\text{K}_3[\text{Cr}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}$ solution was irradiated in ice bath for 2 h. A light green precipitate was created separated by filtration, then washing by using deionized water, ethanol 70% respectively. The product was dried in an oven at 70°C for 24 h and calcined at 700°C for 2 h. A dark green powder was created.

- **For $[\text{Cr}(\text{acac})_3]$**

A (40) ml of $[\text{Cr}(\text{acac})_3]$ solution was irradiated in water bath for 2 h. A light green precipitate was created separated by filtration, then washing by using deionized water, ethanol 70% respectively. The product was dried in an oven at 70°C for 24 h and calcined at 700°C for 2 h. A dark green powder was created.

- For $[\text{Cr}(\text{OC}(\text{NH}_2))_6]\text{Cl}_3$

A (40) ml of $[\text{Cr}(\text{OC}(\text{NH}_2))_6]\text{Cl}_3$ solution was irradiated in ice bath for 2 h. A light green precipitate was created separated by filtration, then washing by using deionized water, ethanol 70% respectively. The product was dried in an oven at 70°C for 24 h and calcined at 700°C for 2 h. A dark green powder was created.

RESULTS AND DISCUSSIONS

Spectral and Structural Properties of Chromium Oxide Samples

FTIR absorption spectra of Cr_2O_3 were offered in figure 2. Metal oxides generally reveal absorption bands below 1000 cm^{-1} arising from inter atomic vibrations. The vibration bands that appears at 626 cm^{-1} , 650 cm^{-1} , 748 cm^{-1} and 771 cm^{-1} respectively attributed to (Cr-O) stretching modes. [26] the absorption bands appears at 1141 cm^{-1} , 1122 cm^{-1} and 1101 cm^{-1} could be attributed to the hydroxyl groups associated with the Cr^{+3} ions. [27]

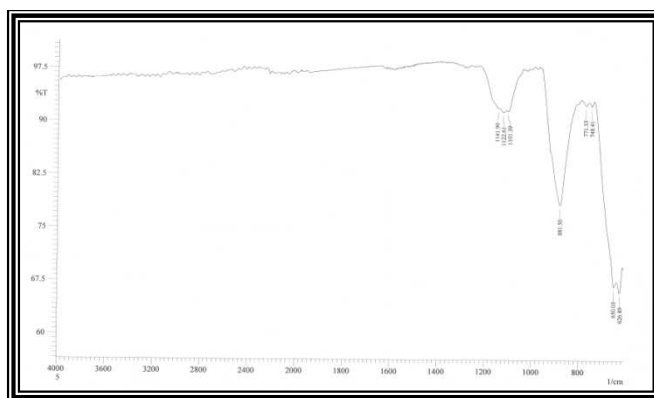


Figure 2: FTIR Spectra for Cr_2O_3

UV-vis absorption spectra for the sample of chromium oxide nanoparticles which have been prepared via photolysis method by using source 125 W appear in the figure 3, this figure shows two absorption bands at 265 nm and 365 nm.

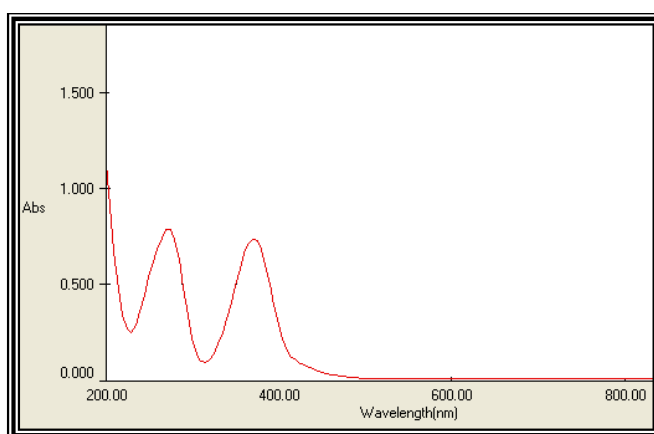


Figure 3: Electronic Spectra for Cr_2O_3

The chemical structure of chromium oxide was studied by XRD with Cu-K α radiation ($\lambda = 1.54056\text{ \AA}$) in the range of 2θ between (10 to 80). Figure 4,5, and 6 show the XRD patterns of the chromium oxide NPs samples which produced by using three complexes via photolysis method, it can be observed that in all cases well (Cr_2O_3 , eskolaite). All

diffraction data were acquired are agreement with (JCPDS card no.00-038-1479), the major peaks were indexed as (012), (104), (110), (113), (202), (024), (116), (211), (122), (214), (300), (119), (220) and (306).^[28]

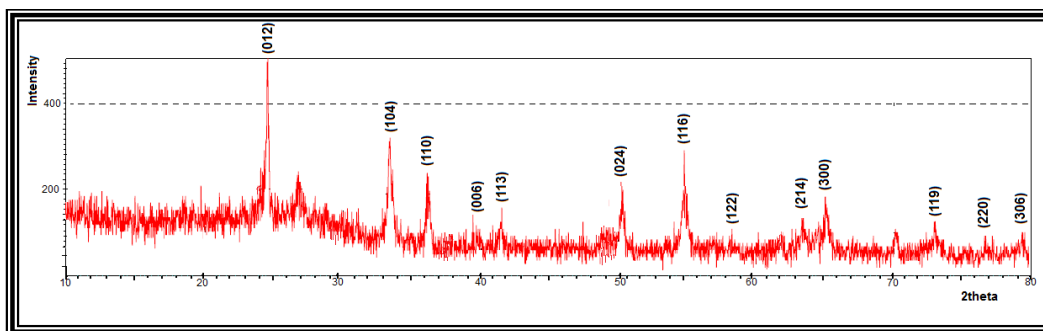


Figure 4: XRD Patterns for Cr_2O_3 Sample Prepared by photolysis $\text{K}_3[\text{Cr}(\text{C}_2\text{O}_4)_3 \cdot 3\text{H}_2\text{O}]$ and Calcined it at 700°C for 2h by Using Source 125 w

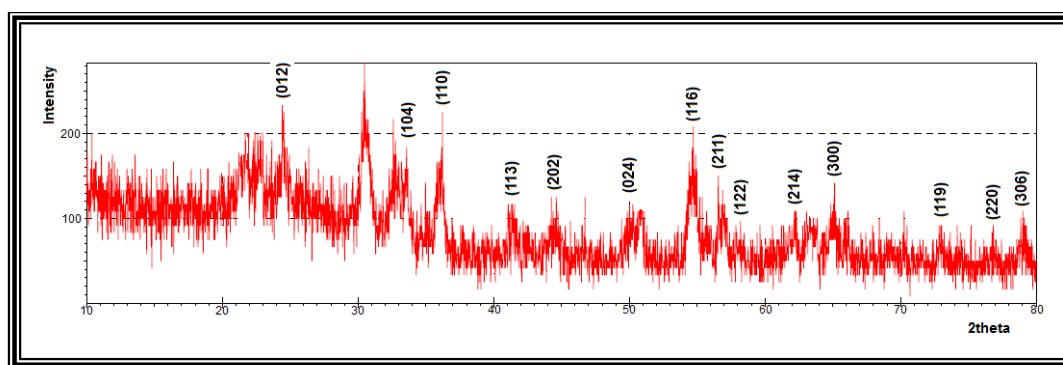


Figure 5: XRD Patterns for Cr_2O_3 Sample Prepared by Photolysis $[\text{Cr}(\text{acac})_3]$ and Calcined it at 700°C for 2h by Using Source 125 w

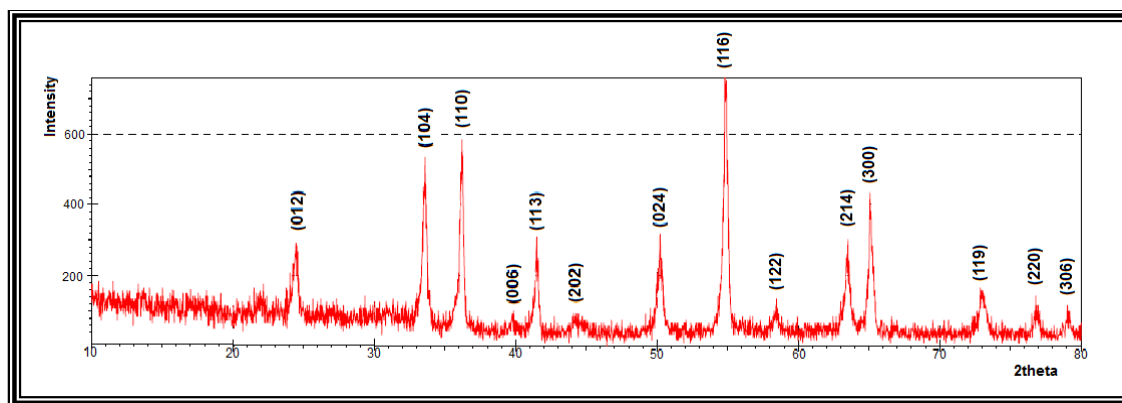


Figure 6: XRD Patterns for Cr_2O_3 Sample Prepared by Photolysis $[\text{Cr}(\text{OC}(\text{NH}_2)_6)_3]\text{Cl}_3$ and Calcined It at 700°C for 2h by Using Source 125 W

Morphology of the Chromium Oxide Particles

The surface morphology of synthesized Cr_2O_3 nanoparticles which have been prepared by photolysis chromium complexes and calcined at 700°C by using source 125 w characterized by scanning electron microscopy (SEM) for three complexes. Through figure 7, 8, and 9 we can observe a large quantity of regular nanoparticles and spherical in shape with average particle size of 40-80 nm.

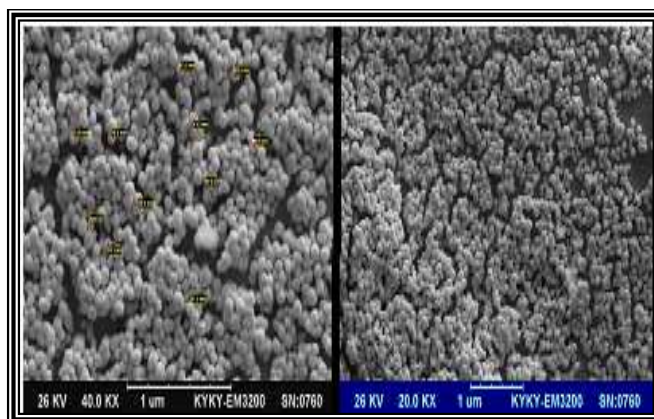


Figure 7: The SEM Image for Cr_2O_3 Sample Prepared by Photolysis $\text{K}_3[\text{Cr}(\text{C}_2\text{O}_4)_3 \cdot 3\text{H}_2\text{O}]$ and Calcined It at 700°C for 2h by Using Source 125 W

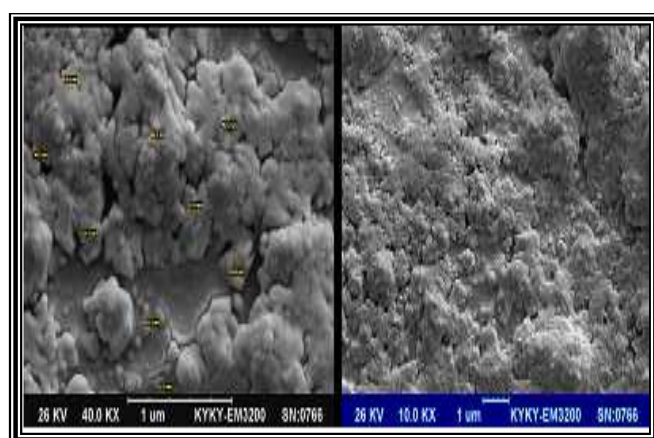


Figure 8: The SEM Image for Cr_2O_3 Sample Prepared by Photolysis $[\text{Cr}(\text{Acac})_3]$ and Calcined It at 700°C For 2h by Using Source 125 W

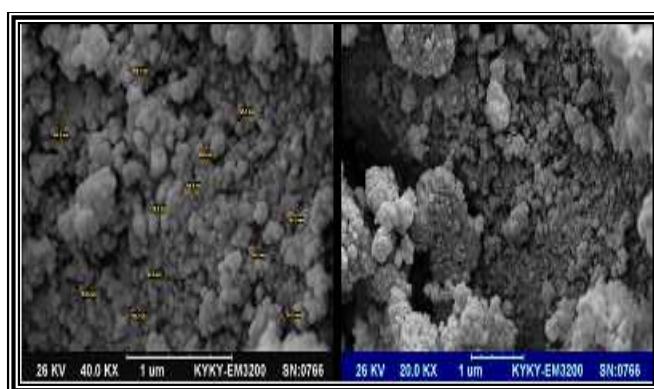


Figure 9: The SEM Image for Cr_2O_3 Sample Prepared by Photolysis $[\text{Cr}(\text{OC}(\text{NH}_2)_6)_3\text{Cl}_3]$ and Calcined it at 700°C for 2h by Using Source 125 W

CONCLUSIONS

In this study, chromium oxide nanoparticles (Cr_2O_3) via photosynthesis method by using three complexes as a source of chromium: $\text{K}_3[\text{Cr}(\text{C}_2\text{O}_4)_3 \cdot 3\text{H}_2\text{O}]$, $[\text{Cr}(\text{acac})_3]$, and $[\text{Cr}(\text{OC}(\text{NH}_2)_6)_3\text{Cl}_3]$. The reaction time was only 2 h without using any surfactant or stirring by using medium pressure lamp with power of 125 watt and calcined the product at 700°C . Thought (SEM) micrographs we can observe a large quantity of regular nanoparticles and spherical in shape and

have a size less than 100 nm. This technique has several features for other techniques of purity, low cost and low particle size .

ACKNOWLEDGEMENTS

I would like to express my thanks to the Head of the Department of Chemistry, Dr.Dhia Hadi Hussain and all the staff of Department of Chemistry-College of Science-AI-Mustansiriyah University.

REFERENCES

1. Zhang L, Webster TJ (2009) *Nano Today* 4:66-80.
2. Mehdi RN, Farhad A, Sorayya H, Neda E, Khadijeh D and Mohammad RG (2016) *Journal of Molecular Liquids* 216, 814-820
3. Aep P, Akito T, and Janusz S (2009) *international journal of hydrogen energy* 34, 3032-3037.
4. Ramin K, and Farideh P (2013) *Journal of NANOSTRUCTURES*. 3, 87-92.
5. R Lazau, C Pacurariu, D Becherescu and R Ianos (2007) *Journal of the European Ceramic Society* 27, 1899-1903.
6. X Pang, K Gao, H Yang, L Qiao, Y Wang and A. A. Volinsky (2007). *Advanced Engineering Materials* 9, No.7, 594-599.
7. U Rothhaar, and H Oechsner (1997) *Thin Solid Films* 302, 266-269 .
8. S Pal, S Kar, S Lastella, A Kumar, R Vajtai, S Talapatra, T Borca-Tasciuc, and P Ajayan (2010) *CA R B O N* 48, 844-853.
9. S Pokhrel, C Simion, V Quemener, N Barsan, and U Weimar (2008) *Sensors and Actuators B* 133, 78-83.
10. C Ramesh, K Mohan kumar, M Senthil, and V Ragunathan (2012) *Archives of Applied Science Research*, 4 (4) 1894-1900.
11. V Teixeira, E Sousa, M Costa, C Nunes, L Rosa, M Carvalho, M Collares-Pereira, E Roman, and J Gago (2001) *Thin Solid Films* 392, 320-326.
12. M Bijker, J Bastiaens, E Draaisma, L de Jong, E Sourty, S Saied, and J Sullivan (2003) *Tribology International* 36, 227-233.
13. M Bijker, J Bastiaens, E Draaisma, L de Jong, E Sourty, S Saied, and J Sullivan (2003) *Tribology International* 36, 227-233.
14. Dr. Ritu (2015) *IOSR Journal of Applied Chemistry (IOSR-JAC) Volume 8, Issue 3 Ver. I. PP 05-11.*
15. Dae-Wook Kim, Seung-Il Shin, Jong-Dae Lee, and Seong-Geun Oh (2004) *Materials Letters* 58, 1894-1898.
16. S El-Sheikh, R Mohamed, and O Fouad (2009) *Journal of Alloys and Compounds* 482, 302-307.
17. T TSUZUKI, and P McCORMICK (2000) *Acta mater.* 48, 2795-2801.
18. N Arul Dhas, Y Koltypin, and A Gedanken (1997) *Chem. Mater.* 9, 3159-3163.
19. Meenambika R, Ramalingom S, and Chithambara T (2014) *Journal of Engineering Research and Applications Vol. 4, Issue 2(Version 3)* 20-23.
20. Z Pei, H Xu, and Y Zhang (2009) *Journal of Alloys and Compounds* 468, L5-L8.
21. D. Vollath, D Szabo, and J Willis (1996) *Materials Letters* 29, 271-279.
22. R Gunnewiek, C Mendes, and R Kiminami (2014) *Materials Letters* 129, 54-56.
23. Jialiang S, Hansong X, Ming G, Han X, and Fusheng P (2014) *Ceramics International* 40, 15051-15055.
24. Tanabe Y. Sugano (1954) *S. J. Phys. Soc. Jpn.* 9, 753.

25. M Salehi, and F Ghasemi (2015) *Journal of Applied Chemistry* Vol. 9, No. 32, 97-100.
26. M Abdullah, F Rajab and S Al-Abbas (2014) *AIP Advances*,4,027121,1-11.
27. El-Sheikh, R., Mohamed, O.,Fouad J. *Alloys Compd.* 482, 302-307.
28. B.D. Cullity, *Elements of X-ray Diffraction*, 2th ed, Addison-Wesley Publishing Company, Reading, 1978, p. 102.

